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(NASA-TM-X-71751) STRUCTURE AND THERMAL
CYCLING STABILITY OF A HAFNIUM MONOCARBIDE
REINFORCED DIRECTIONALLY SOLIDIFIED
COBALT-BASE EUTECTIC ALLOY (NASA) 11 p HC
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**STRUCTURE AND THERMAL CYCLING STABILITY OF A
HAFNIUM MONOCARBIDE REINFORCED DIRECTIONALLY
SOLIDIFIED COBALT-BASE EUTECTIC ALLOY**

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REINFORCED DIRECTIONALLY SOLIDIFIED COBALT-BASE EUTECTIC ALLOY

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ABSTRACT

A nominal composition of Co-15Cr-20Ni-10.5 Hf-0.7 C ("NASA-HAFCO-11") was directionally solidified at 0.8 cm/hr growth rate to produce aligned HfC in a cobalt matrix alloy. The aligned HfC fibers were present as rod and plate types. The diameter of the aligned fibers was about 1 micron, with volume fraction in the range of 11 to 15 percent. The growth direction of the fibers was parallel to the $\langle 100 \rangle$.

The NASA-HAFCO-11 alloy was subjected to thermal cycling between 425° and 1100° C, using a 2.5 minute cycle. No microstructural degradation of the HfC fibers in the alloy was observed after 2500 cycles.

INTRODUCTION

Directionally solidified eutectic (DSE) alloys have received considerable attention for use as turbine blade materials. Their use could permit an increase in metal operating temperatures in advanced gas turbine engines.^{1,2,3} Increasing the turbine inlet temperature would increase efficiency. The increase in efficiency of aircraft gas turbine engines is reflected in a reduced specific fuel consumption or in an increase in thrust per unit weight.

The present day turbine blade materials are nickel-rich solid solution alloys hardened primarily by coherent ordered intermetallic precipitates.⁴ The principal strengthening precipitates, however, tend to dissolve into the matrix above about 1040° C. The dissolution of the precipitates imposes an upper limit on the operating temperature of these conventional superalloys. DSE alloys, currently considered for use, however, are strengthened by strong fibers or platelets directionally aligned parallel to the growth direction in a more ductile matrix. Dissolution of the reinforcing phases is nearly absent even close to their melting point. Many DSE alloys have shown excellent thermal stability during prolonged isothermal exposures.⁵ The thermal stability is attributed to the formation of preferred low energy interfaces during the directional solidification, coupled with the thermodynamic equilibrium of the eutectic phases up to the melting point.

Since the aircraft gas turbine components experience severe thermal cycling, microstructural stability under cyclic thermal exposure is an important consideration in selecting DSE alloys for gas turbine service. The Co-

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base DSE alloys reinforced with tantalum monocarbide, TaC, ("COTAC" alloys), when given an optimum heat treatment,⁶ have better rupture life above about 1020° C than all current superalloys and other DSE alloys. However, the degradation of the TaC fibers and of mechanical properties under cyclic thermal exposure has been found by several investigators.^{7,8,9} The resistance of DSE alloys to thermal cycling damage is affected by the characteristics of coexisting phases;⁵ for example, thermal expansion mismatch, mutual solubility, and phase stability.

Among the transition-metal carbides (TiC, ZrC, HfC, NbC, VC, TaC), the hafnium monocarbide, HfC, is the most stable compound.¹⁰ HfC has the highest melting point of all simple binary compounds,¹¹ about 3890° C. The potential of this HfC reinforced material lies in the fact that creep resistance¹¹ and hardness¹² of the HfC is higher than that of TaC.

The purpose of this investigation was to study HfC as a replacement for TaC as the reinforcing phase in Co-base eutectic alloys. The eutectic temperature of pseudo-binary Co-HfC is reported to be 1400°±20° C, with about 17 w/o HfC content.¹³ This work is concerned with directionally solidified pseudo-binary eutectic of the HfC fiber-reinforced CoCrNi-matrix alloy system, which has been designated NASA-HAFCO-11. The morphology and the crystallographic orientation of the alloy were studied. The microstructural stability of the HfC fibers under cyclic thermal exposure was also examined as a step in determining the potential future use of the alloy as a gas turbine material.

EXPERIMENTAL PROCEDURE

Materials

The purities of raw materials used in this work are shown in Table I. The metals were at least 99.5 w/o pure, except Hf which contained 2.35 w/o Zr. A simple pseudo-binary eutectic of Co-HfC is not suitable for high temperature structural material due to the poor oxidation resistance and the allotropic transformation of the Co-matrix. An addition of Ni was used to stabilize the high temperature FCC phase of the Co-matrix. An addition of Cr was used to give increased resistance to sulfidation and oxidation.

Melting and Directional Solidification

Initial 1.25 kg heats were melted in a 50 kW, 10 kHz induction furnace in calcia-stabilized zirconia crucibles in a partial argon atmosphere. The Co, Ni, and C were melted, followed by the additions of Cr and Hf. The melts were poured into investment molds to make bars of a convenient shape for directional solidification. Each investment mold yielded six cylindrical bars (1 cm diam, 16 cm length).

The bar ingots were remelted and directionally solidified in an alumina tube crucible (I.D. 1.3 cm, O.D. 1.8 cm, length 30 cm) in a modified Bridgman furnace under a flowing argon atmosphere, see Fig. 1. The charged crucible was heated by radiation from a graphite susceptor positioned inside an induction coil. The power was supplied by a 15 kW radio frequency generator operating at 400 kHz. Directional solidification was caused by lowering the crucible through a water spray ring at a constant speed. The temperature gradient at the solid-liquid interface during the freezing process was about 250° C/cm. The melts were maintained at a superheat of about 300° C. No molten metal reaction between the melts and crucibles were observed after the directional solidification process, which took approximately 15 hours.

Metallographic Examination

The directionally solidified bars were polished parallel to the growth direction in order to locate the aligned region. An optical metallographic study was made of the as-polished surfaces of both transverse and longitudinal sections of the aligned region. In order to reveal the carbide morphology, the cobalt matrix was etched away electrolytically in a solution of 30 percent HCl, 7.5 percent CH₃COOH, 7.5 percent HNO₃, and 55 percent H₂O at 12 volts. For examination under scanning electron microscopy, the etched sample was coated with a thin layer of gold-palladium alloy. The measurements of the

volume fraction of the aligned HfC fibers were conducted on scanning electron micrographs of lightly etched transverse sections of the aligned fiber regions.

Crystallographic Study by X-Ray Pole Figure

In order to study the crystallographic orientation of both the aligned HfC fibers and the Co-matrix, an X-ray pole figure technique was used. A sample with a thickness of about 6 mm was taken perpendicular to the growth axis of a directionally solidified sample. The surface of this transverse section was polished prior to obtaining X-ray diffraction patterns. A Norelco X-ray Generator with a Simmons Pole Figure Device was used to determine the pole density of {200} reflection planes. A copper target was used operating at 35 kW and 25 mA. The respective {200} pole densities were plotted as contour lines on a polar stereographic net by selecting appropriate intensity levels. From the constructed pole figures, the crystallographic orientation of the alloy could be determined.

Thermal Cycling

The NASA-HAFCO-7.1 alloy was subjected to thermal cycling in a burner rig apparatus, which was designed to simulate the environment of a jet engine.¹⁴ The alloy was heated quickly to 1100° C in the blast of a gas stream achieved by burning a mixture of JP-5 grade jet fuel and air at Mach 0.3. The alloy was held in the jet for 1 minute, then cooled to 425° C in a blast of room temperature air at Mach 0.7. The total elapsed time for one cycle was 2.5 minutes.

RESULTS AND DISCUSSION

Metallography

Melts of Co-15Cr-20Ni-10.5 Hf-0.7 C were prepared and directionally solidified at a constant growth rate of 0.8 cm/hr. Metallographic examination of polished surfaces, parallel to the growth direction of material, showed several different morphological regions. At the bottom of the DSE bar, a region of blocky excess carbides appeared, while a carbide-depleted dendritic region was observed at the top of the bar. An aligned carbide fiber region formed in the middle of the directionally solidified bars. The length of the aligned fiber region was about two thirds of the total bar length. The density of the aligned region, measured by water displacement, was 8.4 g/cm³. The composition, Co-15Cr-20Ni-10.5 Hf-0.7 C, used in this work, was designated "NASA-HAFCO-11."

Typical microstructures of the aligned HfC fibers in the alloy are shown in Figs. 2, 3, and 4. Figs. 2 and 3 are optical micrographs of unetched surfaces of transverse and longitudinal sections of the directionally solidified sample. Fig. 2 shows uniform distribution of aligned HfC fibers in a section perpendicular to the growth direction, and the alignment of the HfC fibers parallel to the growth direction can be seen in Fig. 3. The measured volume fraction of the HfC fibers in the transverse sections was in the range of 11 to 15 percent. The morphology of the HfC fibers in the alloy can be seen in the scanning electron micrograph (Fig. 4). The matrix was etched away to reveal the aligned HfC fibers. The fibers in the alloy are present as rod and plate types, which look similar to TaC fibers in COTAC alloys.¹⁵ However, one difference in the carbide morphology between the two appears to be the shape of rods. The HfC fibers in the alloy matrix (Co-15Cr-20Ni) tend to have rounded, nonfaceted interfaces as in Fig. 4, compared to the faceted interfaces of TaC fibers as in Fig. 5.

Crystallographic Orientation

Fiber and matrix crystallographic orientations in the alloy were determined using the X-ray pole figure device in the reflection mode from the center of the pole figure out to about 70 degrees. A pole figure is a stereographic projection showing the density of crystallographic poles of selected planes as a function of orientation.¹⁶ The planes selected in both the FCC

structures, HfC and CoCrNi matrix, for the X-ray pole figure were the {200} planes rather than the {111} planes. By plotting {200} pole densities on the stereographic projection planes, one may visualize more clearly the crystallographic orientation. Fig. 6 is a pole figure of {200} reflection planes in the aligned HfC fibers, while Fig. 7 is the pole figure of {200} reflection planes in the Co-matrix. The projection planes are parallel to the sample surface (a section transverse to growth direction). The center of the great circle corresponds to the {100} pole. A strong peak located at the center in Fig. 6 indicates that the aligned HfC fibers have $\langle 100 \rangle$ fiber texture. The growth direction of the fibers are therefore parallel to $\langle 100 \rangle$. The $\langle 100 \rangle$ fiber texture was also reported for TaC fibers in COTAC type alloys.¹⁵ In Fig. 7, several intensity peaks are scattered around the center of the projection. However, there is a tendency for the growth direction of the matrix to be parallel to $\langle 100 \rangle$ direction.

Effect of Thermal Cycling

In evaluating DSE alloys for gas turbine components, the resistance to thermal cycling damage is an important parameter. The NASA-HAFCO-11 alloy was therefore subjected to thermal cycling between 425° and 1100° C, using a 2.5-minute cycle. After 2500 cycles, the microstructure of the HfC fibers in the alloy was examined. The HfC fibers were smooth throughout the thermal cycling with no microstructural degradation as shown in Fig. 8. On the other hand, considerable damage to TaC fibers in Co-base DSE alloys was reported by Dunlevey and Wallace.⁷ Fig. 9 shows the deterioration of TaC fibers in their study as a result of cycling between 427° and 1093° C. The small degree of serration in Fig. 9(a), can be seen after only 200 cycles. The serrations were more fully developed after 2000 cycles (Fig. 9(b)). Fig. 9(c) shows the catastrophic damage to the TaC fibers after 5000 cycles. Dunlevey and Wallace⁷ attributed the formation of serrations to the solubility difference of TaC in the matrix as a function of temperature. At elevated temperatures, the matrix dissolves TaC from the fibers and reprecipitates this material onto the exposed fiber surface at low temperatures. Since the easy growth planes of TaC fibers,¹⁵ {111}, are not coincident with the exposed fiber faces, {110}, the reprecipitating TaC may form {111} oriented ledges on the originally smooth TaC fiber surface.

The thermal expansion coefficient of HfC¹¹ is $6.25 \times 10^{-6}/^{\circ}\text{C}$ in the temperature range from 25° to 1000° C. This is slightly less than that of TaC, which is $6.67 \times 10^{-6}/^{\circ}\text{C}$ in the same temperature range. The slightly greater thermal expansion coefficient mismatch between HfC and the CoCrNi matrix⁸ (about $18 \times 10^{-6}/^{\circ}\text{C}$) might be expected to make the HfC fibers less stable. However, this was not observed here.

The greater stability of HfC, as compared to TaC in the same alloy matrix during the cyclic thermal exposure, may be due to its more negative free energy of formation at all temperatures. At 1100° C, the free energy of formation for HfC¹⁰ is about -52 kcal/gr.at. °C, compared with about -37 kcal/gr.at. °C for TaC. From thermodynamic consideration of single reactive monocarbides, it is likely that the tendency for dissolution of HfC at elevated temperatures in the matrix could be less than that of TaC. However, in the conventional superalloys which usually contain several carbide formers, the order of stability in the monocarbides does not follow exactly the order indicated by free energy considerations.¹⁷

The resistance to thermal cycling damage is known to be affected by solubility change as a function of temperature.⁵ If the process of solution and reprecipitation of HfC over the fiber-matrix interface had occurred during the thermal cycling of NASA-HAFCO-11 alloy, as it occurred with TaC in COTAC alloys,⁷ the HfC fibers should have been covered with serrations. Since in this work no microstructural degradation was observed after thermal cycling (see Figs. 4 and 8), one may conclude that the effect of temperature on the solubility of HfC in the CoCrNi matrix is less than that of TaC in the same alloy matrix.

Walter and Cline¹⁸ considered the shape of aligned fibers on stability of DSE alloys under a prolonged isothermal exposure. They observed that the

faceted Mo fibers in Mo-NiAl were extremely stable, compared with the round nonfaceted Cr fibers in Cr-NiAl. They explained that the faceted fibers are stable since the fiber-matrix interface is constrained to lie in a specific crystallographic plane. However, it has been found in this work that the non-faceted HfC fibers are more stable under cyclic thermal exposure than the faceted TaC fibers in the same alloy matrix.

The melting point of the NASA-HAFCO-11 alloy was estimated to be 1400° C on the basis of temperature measurements at the interface. The melting point of the alloy is somewhat higher than that of COTAC type alloys,⁹ about 1370° C. This higher melting point may also offer a beneficial effect on the thermal cycling stability.

In conclusion, it is proposed that lower solubility change as a function of temperature for the very stable HfC, coupled with its higher melting point, may account for its better microstructural stability than TaC under cyclic thermal exposure.

CONCLUDING REMARKS

In this investigation of the substitution of HfC for TaC as the reinforcing phase in a FCC CoCrNi matrix, a monocarbide reinforced eutectic alloy, NASA-HAFCO-11, was identified that exhibited excellent microstructural stability during cyclic thermal exposure to a temperature of 1100° C. No degradation of HfC fibers in the alloy was observed following these cyclic thermal exposures.

The volume fraction of the HfC fibers in NASA-HAFCO-11 alloy was in the range of 11 to 15 percent. The carbide fiber volume fraction could be varied with the amount of the various alloying elements and by varying growth speed while maintaining the coupled oriented growth.

It is also worthwhile to note that the molten NASA-HAFCO-11 alloy did not have any noticeable reaction with stabilized alumina when exposed at temperatures up to about 1750° C for more than 15 hours of the solidification process.

The mechanical properties of the NASA-HAFCO-11 alloy have yet to be determined. However, the resistance to thermal cycling damage in the alloy suggests considerable potential for the use of this alloy in advanced gas turbine engine components.

SUMMARY OF RESULTS

An alloy of a nominal composition in weight percent of Co-15Cr-20Ni-10.5 Hf-0.7 C composition ("NASA-HAFCO-11") was directionally solidified at 0.8 cm/hr growth rate to produce aligned HfC fibers in a cobalt alloy matrix. The structure and the crystallographic orientation of the aligned alloy was determined, and the alloy was thermally cycled as a step in determining the potential use of the alloy in an advanced gas turbine engine. The following are the major results of this study.

1. The aligned HfC fibers were present as rod and plate types, with the volume fraction in the range of 11 to 15 percent. The surface of the aligned fibers was usually rounded and nonfaceted.

2. The melting point of the alloy was about 1400° C, and the density of the aligned alloy was about 8.4 g/cm³.

3. The growth direction of the HfC fibers was parallel to the $\langle 100 \rangle$ direction. The preferred growth direction of the matrix was also found to be the $\langle 100 \rangle$ direction.

4. No microstructural damage of the aligned HfC fibers was evident in the alloy after thermal cycling between 425° and 1100° C for 2500 cycles, using a 2.5-minute cycle.

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TABLE I. - RAW MATERIALS

| Elements | Purity (w/o) | Form | Supplier |
|----------|-------------------|--------------------|---------------------|
| Co | 99.5 | Electrolytic chips | Kulite |
| Ni | 99.9 | Electrolytic chips | INCO |
| Cr | 99.8 | Electrolytic chips | Union Carbide |
| Hf | 99.8 | Sponge | AMAX |
| | (2.35 Zr include) | | |
| C | 99.5 | Granules BB7 | National Carbon Co. |

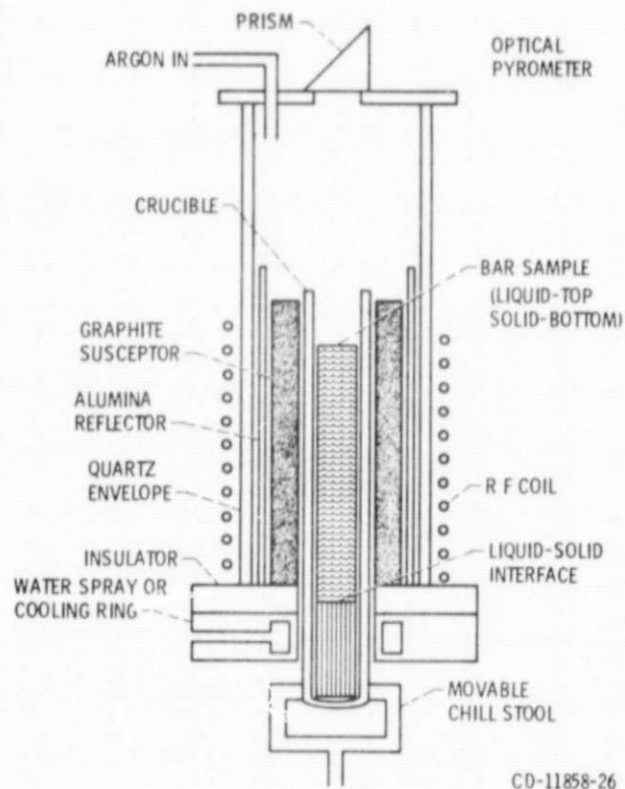


Figure 1. - Directional solidification apparatus.

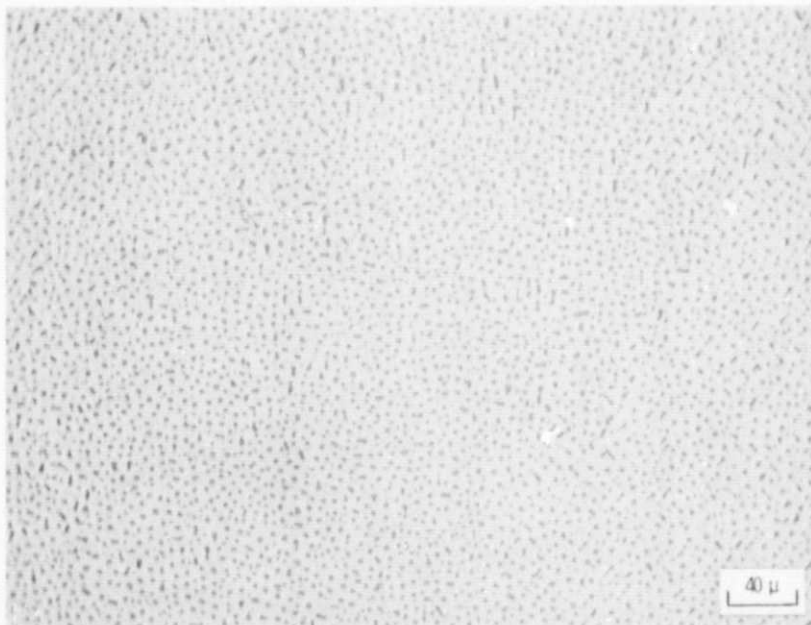


Figure 2. - Optical micrograph of transverse section of directionally solidified Co-15Cr-20Ni-10.5Hf-0.7C. X250.



Figure 3. - Optical micrograph of longitudinal section of directionally solidified Co-15Cr-20Ni-10.5Hf-0.7C. X290.

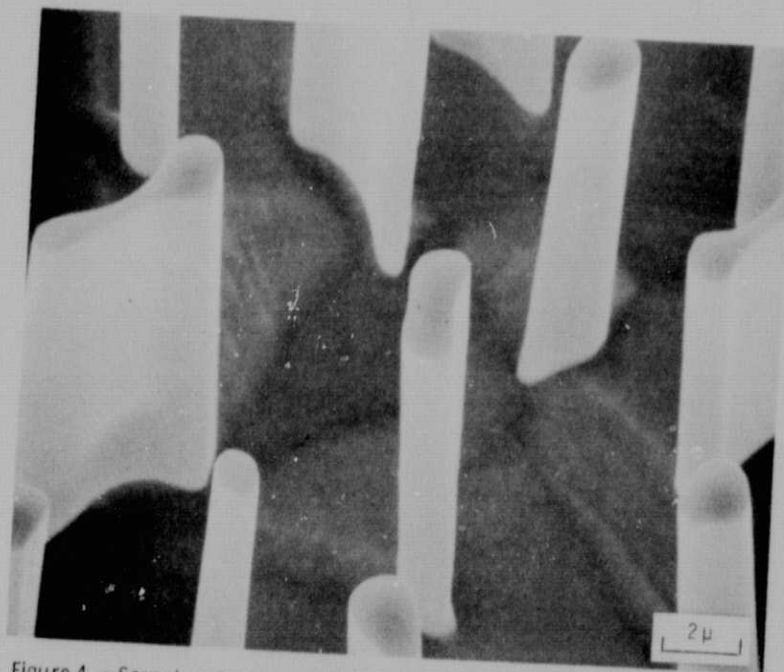
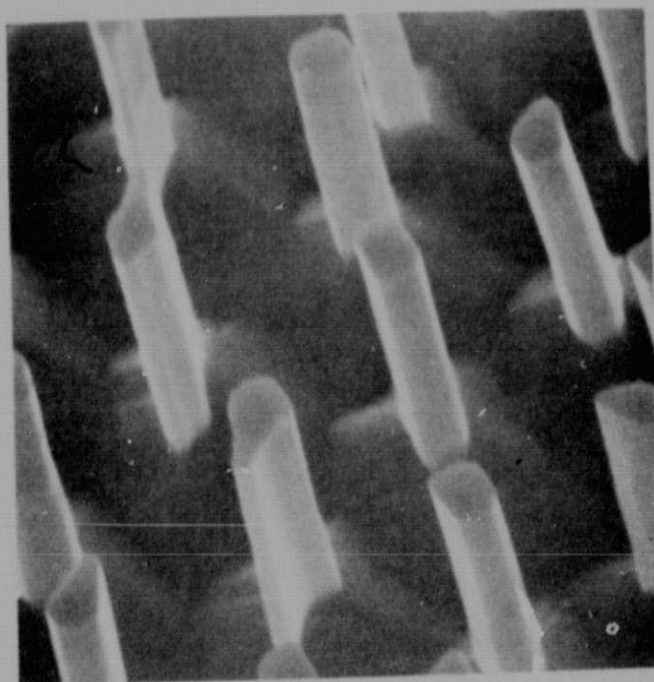


Figure 4. - Scanning electron micrograph of directionally solidified Co-15Cr-20Ni-10.5Hf-0.7C. X5000.



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Figure 5. - Structure of directionally solidified TaC-15Cr-20Ni-Co (after Dunlevey and Wallace, Ref. 7).

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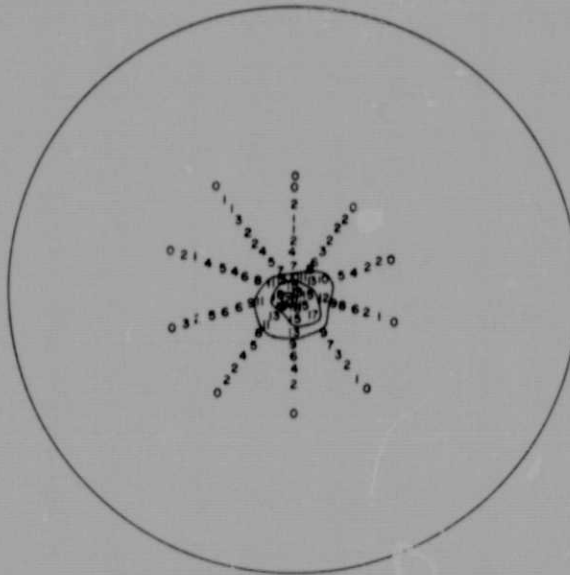


Figure 6. - Pole figure of (200) reflection planes in HfC fibers in Co-15Cr-20Ni-10.5 Hf-0.7 C DS eutectic (projection plane is parallel to transverse section).

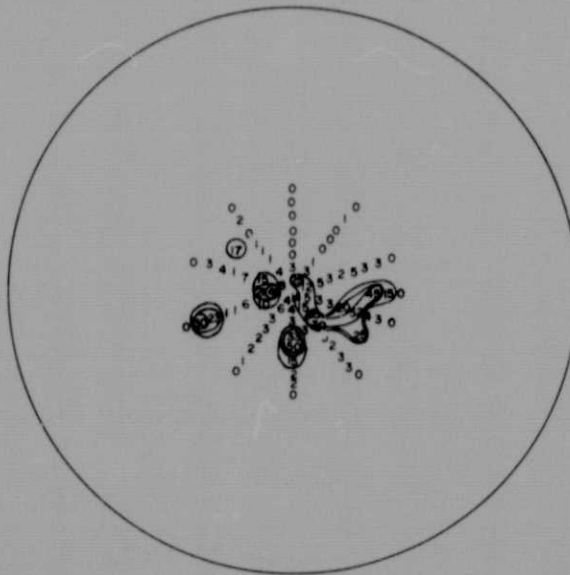


Figure 7. - Pole figure of (200) reflection planes in matrix of Co-15Cr-20Ni-10.5 Hf-0.7 C DS eutectic (projection plane is parallel to transverse section).

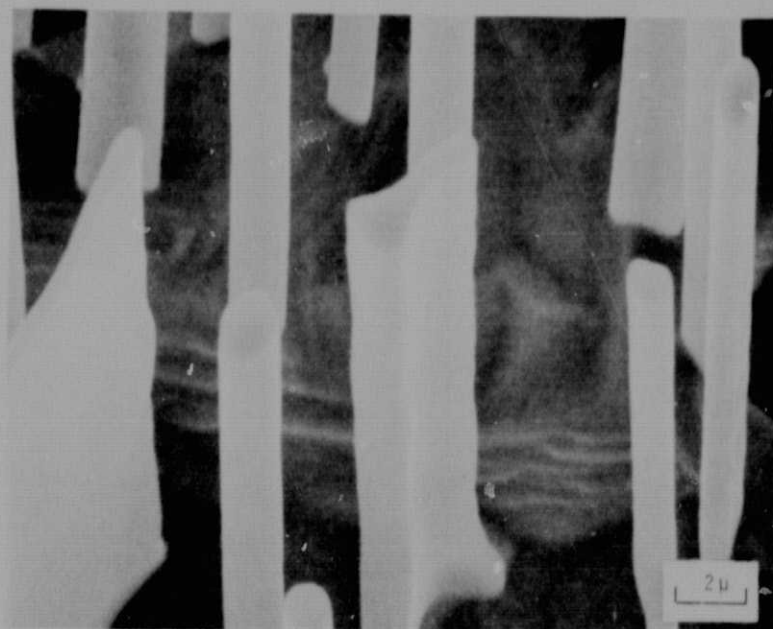
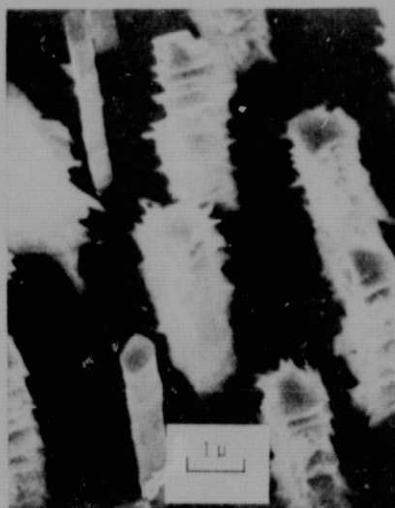


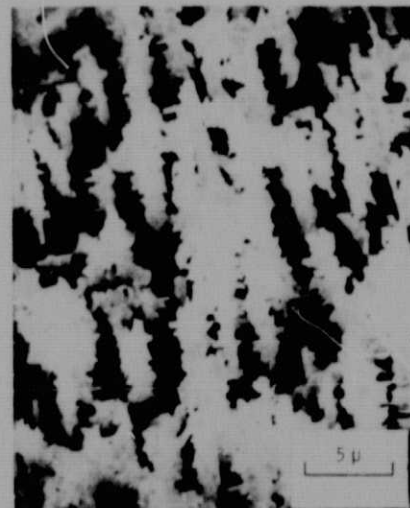
Figure 8. - Microstructure of aligned HfC fibers in Co-15Cr-20Ni-10.5 Hf-0.7C directionally solidified eutectic after thermally cycled between 425° C and 1100° C for 2500 cycles. X2500.



(a) 200 CYCLES.



(b) 2000 CYCLES.



(c) 5000 CYCLES.

Figure 9. - Structure of Co-15Cr-25Ni-TaC cycled between 427° and 1093° C. (After Dunlevey and Wallace Ref. 7).

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